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Fertilizers — Extraction of soluble phosphorus according to Petermann at 65 °C

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Foreword

This document (EN 15921:2011) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by December 2011, and conflicting national standards shall be withdrawn at the latest by December 2011.

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1 Scope

This document specifies the procedure for the determination of soluble phosphorus in alkaline ammonium citrate.

The method is applicable exclusively to precipitated dehydrated dicalcium phosphate ($CaHPO_4 . 2H_2O$).

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 1482-2, *Fertilizers and liming materials — Sampling and sample preparation — Part 2: Sample preparation*

EN 12944-1:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 1: General terms*

EN 12944-2:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 2: Terms relating to fertilizers*

EN 15475, *Fertilizers — Determination of ammoniacal nitrogen*

CEN/TS 15959, *Fertilizers — Determination of extracted phosphorus*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12944-1:1999 and EN 12944-2:1999 apply.

4 Principle

Extraction of phosphorus from the test portion at a temperature of 65 °C with an alkaline solution of ammonium citrate (Petermann's solution) under the specified conditions.

5 Sampling

Sampling is not part of the method specified in this document. A recommended sampling method is given in EN 1482-1.

Sample preparation shall be carried out in accordance with EN 1482-2. Grinding of the laboratory sample is recommended for homogeneity reasons.

6 Reagents

6.1 Water, distilled or demineralized having the same characteristics as distilled water.

6.2 Petermann's solution

6.2.1 Characteristics of the Petermann's solution

— citric acid ($C_6H_8O_7$. H_2O): 173 g/l;

— ammonia: 42 g/l of ammoniacal nitrogen;

— pH between 9,4 and 9,7.

6.2.2 Preparation from diammonium citrate

Dissolve 931 g of diammonium citrate (molecular mass 226,19) in about 3 500 ml of water (6.1), in a 5 l standard flask. Stand in a bath of running water, mix and cool and add in small amounts ammonia. For example, for d_{20} = *0, 906* g/ml corresponding to a level of 20,81 % by mass of ammoniacal nitrogen, it is necessary to use 502 ml of ammonia solution. Adjust the temperature to 20 °C, make up to volume with water (6.1) and mix.

6.2.3 Preparation from citric acid and ammonia

Dissolve 865 g of citric acid monohydrate in about 2 500 ml of water (6.1) in a container of about 5 l capacity. Place the container in an ice bath, and add in small amounts, shaking constantly, ammonia solution using a funnel, the stem of which is immersed in the citric acid solution. For example, for d_{20} = *0, 906* g/ml corresponding to a level of 20,81 % by mass of ammoniacal nitrogen, it is necessary to add 1 114 ml of ammonia solution. Adjust the temperature to 20 °C, transfer to a 5 l standard flask, make up to the mark with water (6.1) and mix.

6.2.4 Checking of the ammoniacal nitrogen content

Transfer 25 ml of the solution into a 250 ml standard flask and make up to volume with water (6.1) and mix. Determine the ammoniacal content on 25 ml of this solution according to EN 15475. If the solution is correct, an amount of 15 ml of c = 0,25 mol/l H_2SO_4 shall be used.

If the strength of ammoniacal nitrogen is greater than 42 g/l, NH_3 can be expelled by a stream of inert gas or by moderate heating to bring back the pH to 9,7. Carry out a second determination.

If the strength of ammoniacal nitrogen is less than 42 g/l, it will be necessary to add a mass M in grams of ammonia solution:

$$M = (42 - n \times 2{,}8) \times \frac{500}{20{,}81} \quad (1)$$

n is the volume of the sulfuric acid c=0,25 mol/l, in millilitres

or a volume, V, at 20 °C:

$$V = \frac{M}{0{,}906} \quad (2)$$

If V is less than 25 ml, add it directly to the 5 l flask with a mass of $V \times$ 0,173 g powdered citric acid.

If V is greater than 25 ml, it will be convenient to prepare a new litre of reagent in the following way.

Weigh 173 g of citric acid. Dissolve it in 500 ml of water. And, taking the precautions indicated, add not more than 225 + $V \times 1\,206$ ml of ammonia solution which was used to prepare the 5 l of reagent. Make up to volume with water and mix.

Mix this volume of 1 l with the 4 975 ml previously prepared.

7 Apparatus

7.1 Common laboratory equipment and glassware, in particular equipment according to 7.2 to 7.4.

7.2 Water bath, which can be maintained at a temperature of (65 ± 1) °C.

7.3 500 ml graduated flask, e.g. Stohmann.

7.4 Dry fluted filter paper, free from phosphates.

8 Procedure

8.1 Test portion

Weigh, to the nearest 0,001 g, 1 g of the prepared laboratory sample and transfer it to the graduated flask (7.3).

8.2 Extraction

Add 200 ml of alkaline ammonium citrate solution (6.2). Stopper the flask and shake vigorously by hand to avoid the formation of lumps and to prevent any adherence of the substance to the sides.

Place the flask in the water bath set at 65 °C (7.2) and shake every 5 min during the first 0,5 h. After each shaking, raise the stopper to equilibrate the pressure. The level of water in the water bath ought to be above the level of solution in the flask. Allow the flask to remain in the water bath a further 1 h at 65 °C and shake every 10 min. Remove the flask, cool to a temperature of about 20 °C, make up to a volume of 500 ml with water. Mix and filter through a dry fluted filter paper (7.4), rejecting the first portion of filtrate. Continue the filtering until a sufficient quantity of filtrate is obtained to carry out the phosphorus determination according to CEN/TS 15959.

Bibliography

[1] EN 1482-1, *Fertilizers and liming materials — Sampling and sample preparation — Part 1: Sampling*

[2] *Regulation (EC) No 2003/2003 of the European Parliament and of the Council of 13 October 2003 relating to fertilisers*, Official Journal L 304, 21/11/2003, P. 0001-0194, Annex IV, method 3.1.5.1

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